

Growth and characterization of germanium nanowires on a flexible aluminium substrate by electron beam evaporation

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Abstract For the first time, Germanium (Ge) nanowires have been grown on a gold (Au) coated flexible aluminum (Al) foil substrate in high vacuum (1×10^{-5} mbar) by electron-beam evaporation of germanium using the vapor-liquid-solid mechanism at a substrate temperature of 380°C. The grown nanowires have been analyzed for their structural, morphological and chemical properties by employing standard techniques X-ray diffraction, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) and X-ray photoelectron spectroscopy, respectively. X-ray diffraction measurements revealed the formation of cubic Ge phase highly oriented in (111) reflection in plane with the Al foil substrate. The morphological observations by SEM have shown the randomly grown nanowires with an average length and diameter of 600 ± 50 and 100 ± 10 nm, respectively, for a deposition time of 30 min. TEM investigation revealed single crystalline nanowires with free of defects. The wettability studies by contact angle measurement have confirmed the hydrophobic nature of the Ge NWs film surface with contact angle for water $110^\circ \pm 1^\circ$. The growth mechanism of Ge nanowires on Al foil substrate has also been discussed.

Keywords Ge nanowires · VLS growth mechanism · Scanning electron microscopy · Flexible substrate · Hydrophobicity · Thin films · e-beam evaporation

Introduction

Crystalline semiconductor nanowires (NWs) have attracted enormous research interest in recent years, owing to their potential use as building blocks for future nanoscale electronic devices (Cui and Lieber 2001). One dimensional (1D) nanostructures of semiconductors such as Si, Ge, GaAs, GaN and InAs have been grown by different physical vapor deposition (PVD) and chemical vapor deposition (CVD) techniques for possible applications in field-effect transistors (FET) (Liang et al. 2007), light emitting diodes (LED) (Qian et al. 2005), Photo Detectors (Ahn and Park 2007) and Solar cells (Tsakalakos et al. 2007). Among these, 1D semiconductor nanostructures, Germanium NWs are of particular interest for high performance nano electronic devices due to their high carrier mobility and low effective mass (Law et al. 2004; Xiang et al. 2006). Ge NWs have been extensively grown by various methods such as pulsed laser ablation (PLD), chemical vapour deposition (CVD) and vapour transport techniques (Wang 2007). Among the techniques, electron beam evaporation (EBE) technique is quite simple in operation to obtain uniform nanowires compared to CVD technique. It is much inexpensive than molecular beam epitaxy (MBE), and being a non-UHV technique, has much higher throughput which makes it interesting for industrial applications. In the literature, there are only few reports on Ge nanowires growth by e-beam evaporation technique (Kumar et al. 2010; Pecorella et al. 2010; Sivakov et al. 2007).

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Flexible substrates such as poly (dimethyl siloxane) (PDMS), polymer substrates, poly (ethylene terephthalate) (PET), thin foils of Al, Cu, have gained increasing attention in the semiconductor industry, due to their light weight and cost in the fabrication process. Ge NWs find applications in fabricating electronic devices (Duan et al. 2003; Lee et al. 2009; Forrest 2004), chemical sensors (Mcalpine et al. 2007). The advantages of Al substrates over Si substrates are flexible, low cost and easy handling. Large area Al substrates are also available commercially. To the best of our knowledge, so far there have been no reports on the growth of Ge NWs on flexible Al substrates by e-beam evaporation. The purpose of depositing on flexible substrate such as Al foil is, to use the Ge NWs as anode for high density Li ion batteries (Chan et al. 2008; Kim et al. 2010). Experiments are under progress to make device fabrication using Ge NWs grown on Al foil substrate.

Water repellence (hydrophobic property) is desirable and important for many industrial and biological processes, such as the prevention of the adhesion of snow to antennae (Nakajima et al. 2001), the prevention of contamination (Coulson et al. 2000), stain-resistant textiles and cell mobility (Barady 1994; Blossey 2003). Hydrophobic surfaces are generally prepared by combining the surface roughness at both the micro and nanoscales with low surface-energy materials. This effect occurs in natural systems quite frequently in the case of a lotus leaf, the self-cleaning process is done by water droplets rolling on the surface and removing dirt and debris. Nature accomplishes this phenomenon through the generation of surface topography that displays structures at micro and nanometre scales. Artificial hydrophobic surfaces have been prepared using various strategies (Coffinier et al. 2007; Furstner et al. 2005; Shiu et al. 2004; Oner and McCarthy 2000).

To our knowledge, there are no reports on wettability studies on germanium nanowires film surfaces. Here, we report for the first time, the growth of pure Ge NWs on Au coated Al foil substrate by e-beam evaporation in high vacuum (1×10^{-5} mbar) with hydrophobic property. The morphology, composition, micro structural and wettability properties of Ge NWs have been studied by SEM, XPS, TEM, XRD, Raman and contact angle measurement, respectively.

Experimental procedure

In the present investigation, to grow Ge NWs by the e-beam evaporation method, pure Ge ingots (purity 99.99% by Blazers) loaded into water-cooled graphite crucible has been used as a source for e-beam evaporation. The rate and

thickness of the deposited Ge has been monitored by a water-cooled quartz crystal monitor. An Al substrate of approximate 160 μm thick has been cleaned in an ultrasonic bath with acetone, and de-ionized water and subsequently, loaded into a sputter coating system and deposited with an Au film of 3 nm thickness.

We have employed BALTEC SCD 500 sputter coating system to deposit gold droplets. For this, after placing the pre-cleaned Al foil substrate in the chamber, the system has been pumped down to high vacuum (1×10^{-5} mbar) and flushed with Ar (99.999%) several times and switch on high voltage for sputtering the gold target. The distance between the substrate and gold target is fixed at 10 cm to obtain uniform gold film.

The Al substrate was heated to the desired temperature by a in-house built substrate heater and the substrate temperature was calibrated prior to deposition by a thermocouple. Au coated Al substrates have been transferred into the e-beam evaporation chamber and was evacuated using a diffusion pump and rotary pump combination. Au coated Al substrates have been annealed in situ at 450°C under a 1×10^{-5} mbar vacuum for 25 min prior to the deposition of Ge to form Au droplets. After annealing, the substrate was cooled to 380°C and Ge has been evaporated by the e-beam at the rate of ~ 0.2 nm/s for 30 min. The applied (emission) current to evaporate germanium is 25 mA and the distance between the source and substrate is 20 cm.

The obtained Ge nanowires on the Al foil substrate has been subjected to adhesion test by using tape. No peeling of the film containing nanowires is observed indicating the good adhesion of the Ge NWs on the Al foil substrate.

The morphological investigation on the Ge NWs has been examined by a scanning electron microscope (Model: SEM FEI Quanta 200). The micro structural properties of the Ge NWs have been evaluated by a transmission electron microscope (Model: TEM, Technai F-30). The structural properties of the films were characterized by X-ray diffraction (XRD, Bruker D8 Advance) and compositional properties were carried out by X-ray Photoelectron spectroscopy (Model: Multilab 2000 Thermo Scientific system) spectroscopy equipped with an Al X-ray source ($h\nu = 1,253.6$ eV). A Phonon confinement study was carried out by Raman spectroscopy (Model: Lab RAM HR Raman system) equipped with a charge coupled device detector (CCD) at working temperature of -70°C . The sample is illuminated by the 514 nm line of an argon ion laser focused at 100 \times objective. All the data have been recorded using 2 mW of laser power and for 20 s of acquisition. The hydrophobic nature of Ge NWs film has been investigated at ambient atmosphere, by contact angle measurement using deionized water.

Results and discussion

SEM

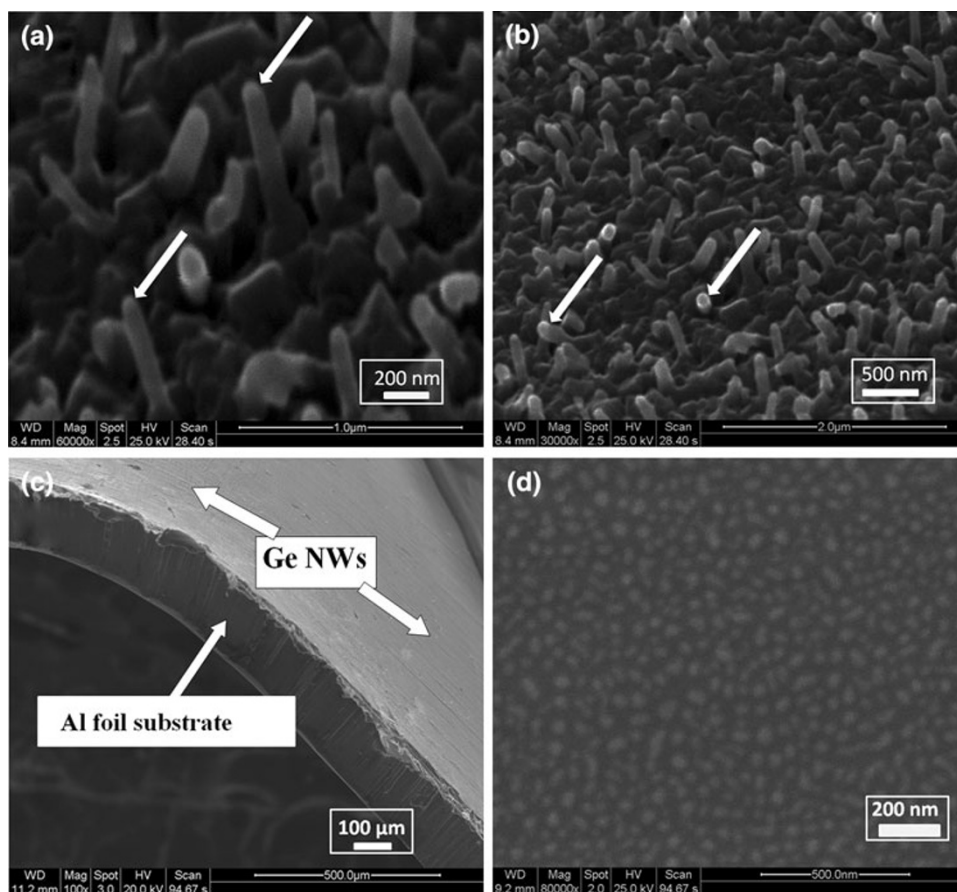
Tilted view of high and low magnification SEM images of the Ge NWs grown on the Au coated Al foil substrate has been shown in Fig. 1a, b, respectively. The average length and diameter of the grown NWs were found to be approximately 600 ± 50 and 100 ± 10 nm, respectively. The obtained Ge NWs have grown randomly, which inhibited us to measure the accurate length of the NWs. From SEM images the real density of the NWs was found to be 3–4 wires/ μm^2 . From Fig. 1a, it is clear that each NW has a gold catalytic particle at its ends and confirming the growth of the NWs following vapor–liquid–solid (VLS) mechanism (Wagner and Ellis 1964). The tip containing the Au droplet is indicated by white arrow mark in Fig. 1a, b. The flexible nature of the Al foil substrate, which contains Ge NWs, can be seen in Fig. 1c. However, we have performed standard tape adhesion test which confirmed good adhesion of the Ge NWs on the surface as well as this is further supported by the Fig. 1c where we have performed measurement on bent sample containing the

substrate where we have not observed any peeling of the film. In case of gold film after tape test, we could see gold droplets uniformly throughout surface of the Al foil. The high magnification SEM image of the gold droplets formed on the Al substrate, after annealing the Au film of 3 nm thickness at 450°C for 25 min has been shown in Fig. 1d. It is noteworthy to mention here that no nanowire growth has been observed on bare Al substrate, eliminating the possibility of catalyst-independent nanowire growth (for brevity SEM images are not shown here).

XRD

The XRD pattern of the Ge NWs grown on an Al substrate has been shown in Fig. 2. The measured reflections have been indexed as (111), (220) and (311) planes of the cubic structure of the Germanium (JCPDS card no. 040545) with a lattice parameter $a = b = c = 5.65 \text{ \AA}$. Apart from the Ge reflections it has been observed for the used substrate Al foil. The diffraction planes (111), (200) and (220) for the aluminium foil substrate with cubic structure (JCPDS no. 851327) have been marked with asterisk. No peaks corresponding to any other impurities or catalytic particles

Fig. 1 Ge NWs grown on Al foil substrate by e-beam evaporation technique. **a** High and **b** low magnification SEM images of Ge nanowires (*tilted view*) (Au tipped on the surface is marked with *white arrows*), **c** the flexible nature of the Al substrate which contains Ge NWs (*bent view*) **d** High magnification SEM image of gold droplets formed on the Al substrate after annealing of gold film of 3 nm thick at 450°C for 25 min



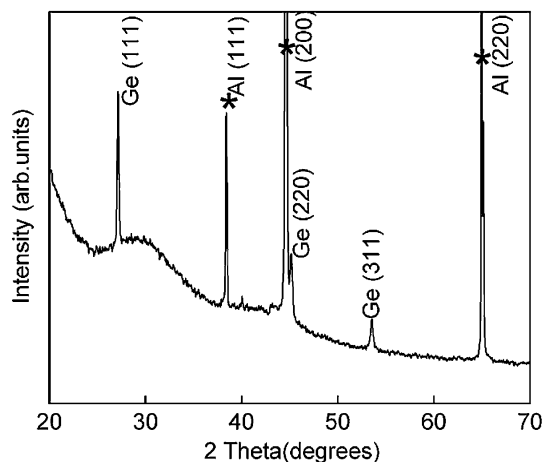


Fig. 2 XRD pattern of the as grown Ge nanowires on flexible Al foil substrate

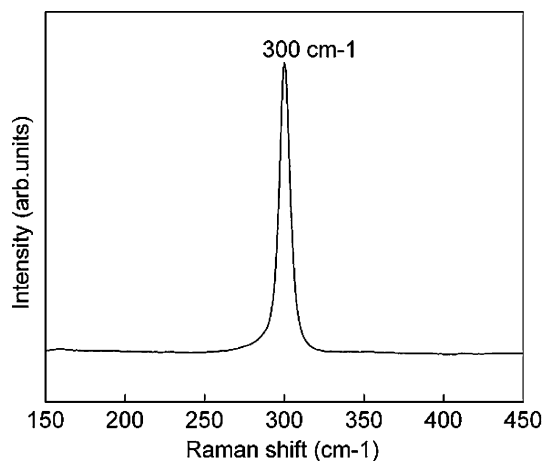
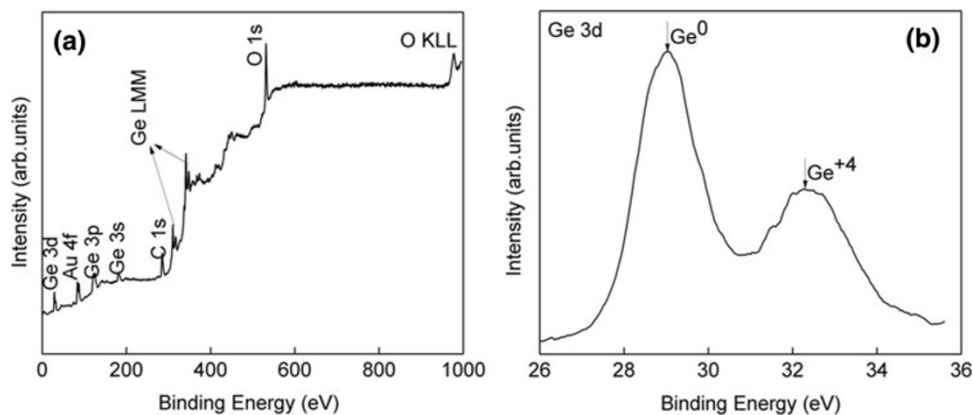


Fig. 3 Raman spectrum of the as grown Ge NWs by e-beam evaporation

were detected. The XRD result confirms that the grown Ge NWs have shown good crystallinity in nature with sharp and high intensity peaks.

Fig. 4 XPS patterns of the as-grown Ge NWs. **a** General survey spectrum **b** High resolution XPS core spectrum of Ge 3d level



Raman

Raman spectroscopy is an important tool for investigating the phonon confinement of nanomaterials, as well as to determine the crystalline nature of materials. The Raman spectrum of the Ge NWs grown on Al foil substrate has been shown in Fig. 3. The sharp peak at 300 cm^{-1} attributed to first order optical phonon contribution of the Ge–Ge stretching mode (Pei et al. 2010; Jalilian et al. 2006), and also confirmed the crystalline nature of the Ge NWs, which further supports the XRD measurement. The peak at 300 cm^{-1} of the NWs is red shift when compared to the bulk Ge (at 302 cm^{-1}) (Jin et al. 1999). The minimum size of the nanowire was observed to be $\sim 100\text{ nm}$. With this size, no quantum confinement can be observed. The phonon confinement effect can cause an insignificant shift of the peak to the lower energy. Therefore, the red shift of the Ge–Ge Raman stretching peak can be understood in terms of the phonon confinement effect (Pei et al. 2010). However, more investigation in this direction is under progress.

XPS

X-ray photoelectron spectroscopy measurements were performed on the Ge NWs, grown on an Au coated Al foil substrate. The XPS analysis of the Ge nanowires has been surveyed in the binding energy (BE) range of 0–1,000 eV has been shown in the Fig. 4a. No contamination species were observed within the sensitivity of the instrument apart from the adsorbed atmospheric carbon and Auger peaks of Ge LMM and O KLL. The binding energy calibration of the spectra has been referred to a carbon 1 s peak located at 284.54 eV.

Figure 4b shows the XPS spectra of Ge 3d core level, obtained from the GeNWs. The core level Ge 3d spectrum indicates the binding energy of Ge^0 at 29.05 eV and that of Ge^{+4} at 32.25 eV, respectively. This observation is in good agreement with the XPS database (Castain 1992). The presence of a Ge^0 peak implies that the main body of the

Ge NWs is composed of pure Ge. It is evident that, there is a slight formation of the GeO_2 (32.25 eV) on the surface of the formed Ge NWs, and this may be due to exposure of samples to atmosphere (Molle et al. 2006).

Wettability

Wettability is a very important property of solid surfaces and is governed by the chemical composition, roughness, the manner of the surface preparation and the surface cleanness. The wettability property was characterised by the contact angle water to the solid surface. The formation of a contact angle at the solid–liquid–gas boundary on a rough surface can be described by two models namely Wenzel's equation (Wenzel 1936) and Cassie and Baxter's law (Cassie and Baxter 1944). A hydrophilic surface has a contact angle less than 90° . If the value of the contact angle is greater than 90° , then the surface is hydrophobic and if the value of the contact angle is more than the 150° , then the surface is super hydrophobic. However, to the best of our knowledge there is no report on the wettability studies on Ge NW films. Figure 5 shows the photograph of the water droplet on the Ge NWs film grown on Au coated Al foil substrate. The measured contact angle was $110^\circ \pm 1^\circ$ for Ge NWs film surface. This confirms that the NWs film surface is hydrophobic in nature. Hydrophobic nature of the surface was caused by air trapped in the rough surface, this being obtained by combining micron and nanometer-scale structures induced by nanowires (Coffinier et al. 2007; Cassie and Baxter 1944). Even though Ge itself is hydrophobic, one expects same for Ge NWs films but we have given experimental evidence to the scientific community that Ge NWs film surface showing hydrophobic nature. However, there is no report as on today on hydrophobicity ($110^\circ \pm 1^\circ$) of Ge NWs grown by e-beam technique or any other PVD or CVD technique. This is only a preliminary experiment as our aim to grow vertical Ge NWs with improved wettability towards super-

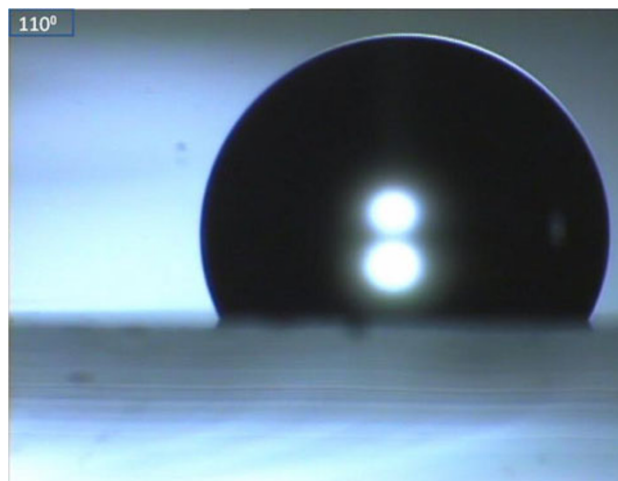


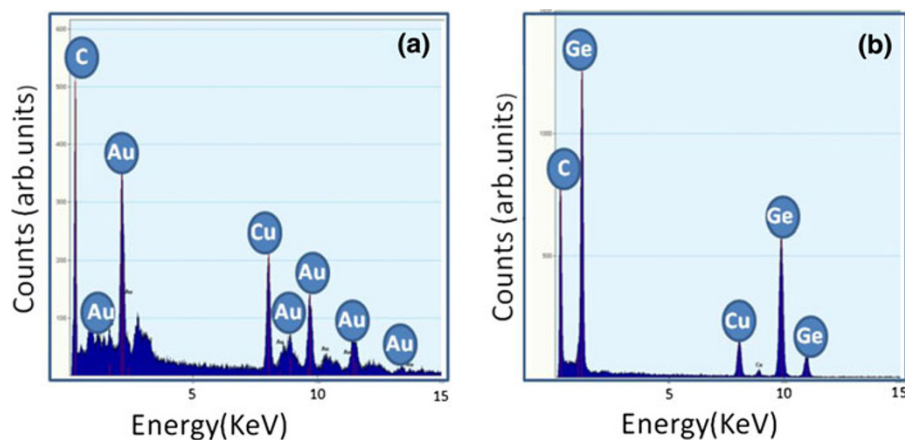
Fig. 5 Photograph of water droplet on Ge NWs surface

hydrophobicity. In fact, there are some references where investigators have modified the surface of Si NWs by coating some additives (Yung and Gau 2010). Experiments are under progress to increase the contact angle with different growth conditions of Ge NWs.

TEM-EDX

The Ge NWs were scraped from the Al foil substrate and moved on to a Cu grid for TEM characterization. Energy dispersive X-ray analysis has been performed on the tip of the Ge nanowire as well as on the lateral part of the wire which has been depicted in Fig. 6a and Fig. 6b, respectively. Figure 6a it is revealed that the tip of the nanowire contains gold only (Kumar et al. 2010; Sutter and Sutter 2008), whereas the base of the NW contain Ge only. Further, no other impurities or other catalytic particles were observed on the grown NW during TEM analysis. The inset (top left) of Fig. 7 shows the high magnification TEM image of the single Ge NW. This clearly shows the Ge NW with an Au nanoparticle at its end (indicated by white

Fig. 6 Energy Dispersive X-Ray spectrum of **a** Tip of the nanowire, **b** rest of the nanowire



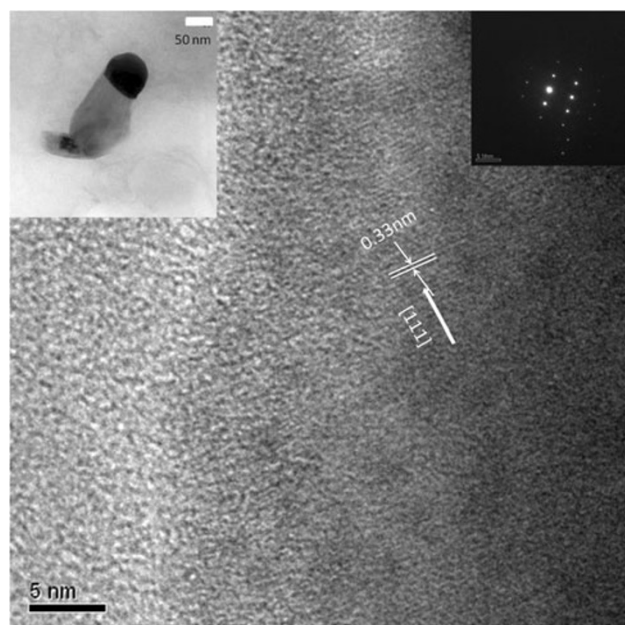


Fig. 7 High Resolution Transmission Electron Microscopy (HRTEM) image of a single germanium nanowire *inset top left* TEM image of single nanowire with an Au nanoparticle at its end *inset top right* selected area electron diffraction (SAED) pattern for Ge NW

arrow). The substrate temperature maintained well above the eutectic temperature during growth of NWs, this further confirmed VLS growth of the Ge NWs. The HRTEM image shows the single crystalline nature of the Ge NWs and the nanowire is free of defects. The inset (top right) of Fig. 7 shows the selected area electron diffraction pattern recorded on the NW, which is also supporting the single crystalline nature of the NW. Nanowires have been grown on Au coated Al foil substrate having different orientations with respect to substrate, so that there may be different growth directions of nanowires. The lattice spacing in HRTEM in Fig. 7 is found to be 0.33 nm, which corresponds to the “d” spacing of the (111) plane of Ge. The obtained results are in good agreement with observed XRD measurements.

Growth mechanism

Germanium NWs were grown both perpendicular and inclined with respect to the substrate as shown in the SEM images (Fig. 1a, b). The growth mechanism of Ge NWs grown by e-beam evaporation is similar to the mechanism proposed for Ge, Si NWs grown by e-beam evaporation (Pecorella et al. 2010; Irrera et al. 2009). The growth of NWs can be explained in terms of two competing processes: germanium atoms directly impinging on the gold droplet and germanium atoms adsorbing on the substrate. Germanium atoms impinging on the gold droplet form an

alloy with the gold droplet, and causing super saturation of alloy results in a growth of germanium nanowire occurred just at the liquid/solid interface of the alloy and the substrate. This process results in the growth of NWs with a gold particle at the end of the each wire. This mechanism clearly shows the VLS mechanism. Atoms impinging on the substrate also have a fundamental role in the axial growth; in fact, growth conditions allow them to easily diffuse over the sample in such a way that some of the atoms can diffuse along the growing Ge sidewalls. These atoms can be adsorbed at the solid germanium/liquid eutectic interface under the gold droplet. Therefore, they may be having an important contribution to the NWs growth. To our knowledge, as on today in the literature, the maximum length grown with molecular beam epitaxy, electron beam evaporation methods is about 2 micrometer (Pecorella et al. 2010). The main reason for not getting lengthy nanowires is adatom diffusion length of growth species. It plays a key role in nanowires growth by EBE (Artoni et al. 2011; Xu et al. 2011). Initial stage of nanowire growth depends on growth species which deposit on the substrate contribute to nanowire growth. When length becomes larger than the diffusion length, then the length of the nanowire depends mainly on the diffusion of adatoms fall that adsorb directly onto the NW side walls or on the catalyst particle surface. Therefore, after few nm nanowire growth, growth become slow and at the same time 2D layer deposit increases on the substrate, and nanowire grows by collecting growth species from the sidewalls and catalyst. If we increase time of deposition gold diffuse away from the original seed particle, as a result, the nanowire cannot grow in lengthy any more, but over growth on the side walls occurs.

Conclusion

For the first time, Ge nanowires have been successfully grown on a flexible Al foil substrate by e-beam evaporation technique under high vacuum (1×10^{-5} mbar) at 380°C substrate temperature using Au as a catalyst. SEM measurements indicated that the grown Ge NWs have been oriented in a random direction with respect to the substrate. XRD and Raman studies revealed the crystalline nature of the NWs and also the phonon confinement effect in Ge NWs. XPS studies revealed that the grown NWs are bit oxidised due to exposure of the samples to the atmosphere. From the TEM studies, the growth of the NWs follows the VLS mechanism. The as grown nanowires were single crystalline in nature and free of defects. Contact angle measurement revealed the hydrophobic nature of the Ge NWs film surface. Experiments are under progress to grow the Ge NWs in vertical direction perpendicular to the

substrate with minimum length of 1 micron for practical applications in high speed electronics, high density Li ion batteries, low k dielectric materials.

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